# Glow Discharge Cleaning for the Main Injector

### 1 Introduction

To minimize beam losses due to beam-gas interaction, the vacuum system of the Main Injector is required to obtain a pressure of 5×10<sup>-9</sup> Torr. The low pressure is the result of the average outgassing rates which can be routinely obtained in several egineering means, such as vacuum degreassing, vacuum fire at 1000°C, glow discharge cleaning, and bake out, etc. However, during accelarating proton beams, the residual gas molecules can be ionized by the circulating protons. These ions are accelerated by the beam-wall potential and bombard the walls of the vacuum chamber. This ion bombardment desorbes gas still adsorbed on the wall even after 300 °C bake out. The pressure increases the ion bombardment. This can result in a runway process giving a rise in pressure and consequent rapid beam loss. Analysis of one of these called pressure bumps revealed that the major component of the beam induced pressure bump as observed at a proton accelerator was CO (≥ 50%) which came from carbon contamination of steel and/or surface adsorbed CO molecules. The vacuum instability caused by this ion desorption determines the maximum beam current which can be obtained.

C from the Auger spectra appears to be in the form of graphite which is non-volatile and is therefore not completely removed by means of bake out. Glow Discharge Cleaning which can strongly remove carbon from the surface of vacuum systems may create a 'wall pumping speed' for CO. After an ion dose  $10^{18-19}$  ions/cm² the integrated surface impurities, such as CO, CO<sub>2</sub> and H<sub>2</sub>O, can be removed from stainless steel treated by Ar, Ar/O<sub>2</sub> or H<sub>2</sub> glow discharge. This minimizes the ion-induced molecular desorption and hence increases the vacuum stability. Table 1 summarizes the results of some laboratory tests on the efficiency of glow discharge cleaning on stainless steel. The desorption yield for CO, the important parameter for the stability of proton accelerators and storage rings, is reduced to  $\leq 0.01$ .

Table 1 CO desorption yield in molecules/ions from stainless steel

standard cleaning	10
bake out 300°C 24 hr	1.8
discharge 10 <sup>18</sup> ions/cm <sup>2</sup>	≤ 0.01
air 12 hr 1 bar	5
bake out 300°C 24hr	0.9

A.G.Mathewson, CERN-ISR-VA/76-5

Upon air exposure of a treated vessel, only a few monolayers of oxygen and carbon readsorb, therefore, justifying the benifits of the technique even when applied as an one-time only procedure prior to vessel assembly. Therefore GDC technique can be applied either as an one time only surface treatment prior to vessel assembly or incorporated as a routine in situ conditioning technique. In either application GDC treatments with a variety of reactant gases are shown to reduce particle induce of desorption coeffecients and thus reduce beam conditioning time.

# 2 Description of glow discharge

The glow discharge has been described in many studies of gaseous electronics. It occurs under specific gas pressure and current flows through gas excitation and formation of plasma conditions. Deexcitation of the gas molecules produces visible light. Ions can sputter adsorbed molecules or atoms at the cathode surface (vessel wall) resulting in surface cleaning. The exitation source can be a D.C., a R.F., a microwave power supply or in some case a parrallel combination of D.C. and high frequency sources.

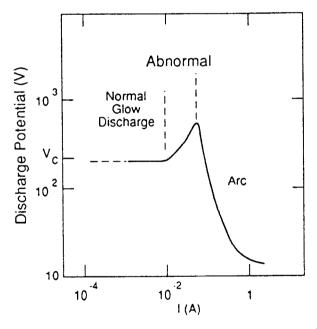


Fig. 1. Voltage (V) - Current (I) characteristics of the classical dc glow discharge

The basic glow discharge regimes are often illustrated in terms of the I-V characteristic (Fig.1) of a gas discharge between two electrodes. A glow discharge may be initiated at a low current appropriate to the "normal glow discharge" regime where the cathode fall potential is independent of the discharge current. A glow discharge can not be sustained below a minimum current density to the cathode, typically  $\sim 1 \mu A/cm^2$ , which is necessary to sustain the discharge by secondary electron emission. The more useful regime for GDC is the "abnormal glow discharge" where Ve is proportional to the discharge current since large current and thus larger vessel surface ion fluxes can be sustained. However, the discharge current can not be increased to arbitrarily large values. When electrode current densities exceed 100µA/cm<sup>2</sup>, discharge makes a transition to the arc regime, which is totally inappropriate for chamber conditioning because of deposition of electrode materials. The typical range of available cathode current density for the D.C. driven abnormal glow discharge is of the order of 20-100  $\mu$ A/cm<sup>2</sup>, or equivalently, singly ionized wall fluxes of  $1\times10^{14}$  -  $5\times10^{14}$ ions/cm<sup>2</sup>-s. The basic parameters of D.C. GDC recipe are listed in the following table:

Table 2 The basic parameters of GDC

Pressure	Torr	2×10 <sup>-2</sup>
Voltage	V	~350
Gas flow	c.c/s(STP)	~0.2
Current density	$\mu A/cm^2$	20-100
Ion dose	ions/cm <sup>2</sup>	10 <sup>18</sup>
Wall temperature	°C	200-350

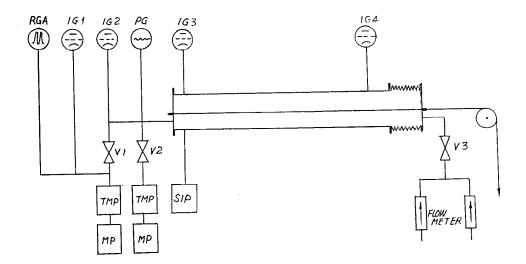
The early laboratory studies of the glow discharge cleaning emphasized noble gas discharge. The GDC technique was given its first wide exposure to the vacuum technology community with the development of Argon/10%Oxygen GDC techniques for the surface treatment. Argon/10%Oxygen plasma was found to be more effective than pure Argon plasma in removing carbon, because of the high surface carbon removal rates that could be generated in such discharge through the formation of CO and CO<sub>2</sub> reaction products which would be then removed by the pumping system. The experiments shown that the desorption yield of CO with Ar/10%O<sub>2</sub> was approximately three times larger than that with pure argon, and oxygen was almost completely consumed.

## 3 Experimental arrangement for beam pipes

The simplest arrangement for the glow discharge cleaning is a D.C. system where the beam pipe wall should be grounded as the low potential side of the exitation source. This arrangement subjects the beam pipe wall to the ion bombardment which removes surface and near surface impurities from the vessel.

Fig.2 shows the schematic diagram of the experimental arrangement for beam pipes, in which, TMP, MP and RGA stand for the turbo-molecular pump and mechanical pump, and the residual gas analyzer respectively.

Fig.2 Schematic diagram of GDC setting up for beam pipes



The system is constructed in a beam pipe being cleaned and pumped with an  $\sim 100 \text{ l/s}$  turbo-molecular pump via a known limiting conductance, for example 3 l/s for N<sub>2</sub>. Argon/10%Oxygen mixture is blown in the pipe from the end through a metering valve to maintain the required pressure. The fresh supply of oxygen is necessary for effective removal of carbon (CO and CO<sub>2</sub>). Because the argon glow discharge operate at  $2 \times 10^{-2}$  Torr with a high ion density, the total pressure is measured with a thermocouple gauge or Pirani gauge placed outside the discharge area. A residual gas analyzer works at  $\sim 10^{-6}$  Torr in an adjecent turbo-molecular system connecting to the beam pipe by a leak valve. The glow discharge operation may be carried out with the discharge chamber at any temperature between 20 - 300°C.

In order to have uniform glow discharge cleaning along the pipe length, a 0.5

mm wire diam tungsten or molybdenum wire is positioned through the center of the pipe and serves as an anode. The wire could be used as a heating element by an isolation transformer during bake out. With about 7 A current, the wire will be able to reach high temperature and provide radiant heat to the 6 m long beam pipe. Bake out temperature of 200 °C could be reached within 4 hours.

The electrode is powered by an electric exitation source which produces glow discharge. The exitation source could be a D.C. power supply with a capacity of 1 kVA, a voltage of 500 V. The power electrode can take the full current of the discharge without over heating. In the D.C. power supply system, a load resistor is usually put in series with the anode, unless a current limiting power supply is used in order to limit the current drawn by unipolar arcs which are quite common initial stage of glow discharge treatment of dirty surface.

### 4 Measurements of GDC

To effectively clean the beam pipes in the short time, the optimum glow discharge condition must be known. These include the voltage-current characteristics, the necessary throughout and pumping speed at selected discharge current to supply a fresh plasma, and the effective monitoring of the exhaust. The voltage-current characteristics at different pressure must be known to identify the regime of self-sustaining glow discharge in a pipe. The Ar/10% Oxygen mixsture is introduced into the system and the D.C. voltage is applied to initiate the glow. The uniformity of the glow discharge along the pipe is checked visually and by measuring the return current through vacuum chamber. No stable glow is observed at pressure less than  $2 \times 10^{-3}$  Torr or greater than  $2 \times 10^{-1}$  Torr. The low current density limit is usually of the order of 1  $\mu$ A/cm<sup>2</sup> and is determined by the minimal current necessary to sustain the discharge by electron impact ionization within the volume of the discharge. Under current density lower than  $1\mu A/cm^2$  the glow discharge will become intermittent. The maximum current density is typically of the order of 100  $\mu$ A/cm<sup>2</sup> and it is limited by excessive electrode heating or/and transition of the glow discharge to the arc regime. Both the high current and low current density limits are dependent on the gas pressure and electrode materials.

Current density of 20  $\mu$ A/cm<sup>2</sup> (about 300 mA for the whole pipe) and pressure  $2\times10^{-2}$  Torr are selected for the most measurements which is a compromise between the available throughput and the required time span for cleaning. The energy of ions is to close the anode cathode potential which is approximately 350 V. Higher ion energy gives higher desorption yield, but also increases implantation and tapping of the ions the wall.

The throughput and the effective pumping speed at different metering valve

settings are measured by monitoring the rate of pressure rise. At an average pressure of  $2\times10^{-2}$  Torr the throughput and effective pumping speed are estimated to be approximately 3.6 STP c.c./min and 3 l/s respectively. At a discharge density of  $20~\mu\text{A/cm}^2$ , which is about half the ion equivalent of the throughput, most desorbed molecules are swept out before setting ionized and resputtered on the surface and fresh plasma is ensured.

The RGA as a monitor for surface cleanliness measures the composition of the glow discharge exhaust from which the deposition yield (net increase of molecules per unit discharge in the exhaust) can be derived. To determine quatitatively the partial pressure of each gas in the plasma exhaust, the sensitivities and cracking patterns of the RGA for different gases have to be known and the changies in the ratio of partial pressure before and after leak valve are needed. The RGA should be calibrated against a standard mixture to obtain the sensitivities and cracking patterns for different gases. The background is measured by turning of the discharge power from time to time and recording the partial pressure.

The desorption yield rate can be calculated by the matching the gas flow and by the derived RGA correction factors.

## 5 In situ GDC processing

The vacuum requirement of the Main Injector is very critical in the area of vacuum techniques. Cleaned components and devices have to expose to air during the assembling and installation. After atmospheric exposure, even if done under well controlled venting with dry nitrogen, the desorption yield increases to almost its original value. It is unavoidable to prevent cross contamination, and a significant part of the cleaning effect may be lost. As a large scale vacuum system of the Main Injector, it would be very difficult to keep cleanliness of surfaces after pre-treatments for a long time.

In situ bake out and glow discharge cleaning are widely used for accelerators and storage rings. In the case of in situ treatment, complicated surface pretreatment procedures may be ignorable. In order to bake out the beam pipe, the magnet gap must be enlarged for using thermal insulations. This would increase the production and operation cost of magnets. Whereas in situ GDC techniques have a obvious benefit of minimizing conditioning time after the system installed or air inrushed. It requires only standard vacuum equipments, in particular it can be done any time and anywhere. However, the application of D.C. GDC in the accelerator tunnel might pose some difficulties of the removal of electrodes from beam pipes after conditioning. Because the added complication to an accelerator is the requirement of distributed anodes within

the chamber which do not interfere with the beam aperture, but are capable of sustaining a quasi-uniform discharge throughout the entire chamber.

Electron Cyclotron Resonance (ECR) glow discharge cleaning, electrode-less glow discharge, is considered to be worth using for the Main Injector. It has an advantage of lower metallic sputtering because of the low plasma potentials. A microwave generator with a frequency of 1-3 GHz and a power capacity of about 500 w, and an additional magnetic field of 800-1 200 G are needed to the excitation volume. Fortunately, about 70% of 3.3 km circumference of the ring is inside magnets, the ECR glow discharge cleaning can be conveniently used for the Main Injector. It only requires an additional port on stubs of the beam pipe, placing a short monopole antenna which do not interfere with the beam aperture, but is capable of sustaining an uniform discharge through entire beam pipes.

# 6 Procedure of glow discharge cleaning

A general procedure is as follows:

## a) Chemical cleaning;

Before discharge cleaning, all components and beam pipes must be chemically cleaned to remove the cutting oils, dust, fingerprints, etc. Since it is the tightly bound surface gas, not removed by the bake out, which is desorbed by the ions or electrons, one criterian for choice of the chemical pre-cleaning method is that it leaves the vacuum chamber surface with as little desorbable gas as possible.

### b) Pump-down 2 days;

In order to reduce background outgassing rate as low as possible, the beam pipe should evacuate at least 2 days before the glow discharge cleaning.

## c) Bake out 200°C for 24 hours;

Vacuum bake out before glow discharge has been found to be more effective since most loosly bounded molecules (CH<sub>4</sub>, H<sub>2</sub>O, N<sub>2</sub>...) are removed thermally.

## d) GDC with Argon/10% Oxygen;

An atomically clean surface can be obtained after an accumulated dose (product of discharge current density and discharge time) of  $10^{18}$ - $10^{19}$  ions/cm<sup>2</sup>. It will be reached within 4-6 hours depending on the discharge current.

### e) Bake out 200°C to remove the implanted Argon;

Since the ion energy is of the order of 300 eV there will be some argon implanted into the discharge cleaned vacuum vessel walls from which it will thermally outgas and provide an undesirable source of argon. Therefore after argon glow

discharge the thermal outgassing rate of argon at about  $200^{\circ}$ C is about  $\sim 10^{-10}$  Torr l/s cm<sup>2</sup>. The beam pipe being tested should be pumped contineusly for 24 hrs. In this case the residual argon thermal outgassing rate at room temperature will be negligible.

### 7 Conclusion

In order to limit the outgassing of beam pipes to a minimum, and provide a stable vacuum condition for the Main Injector, we have some choices to follow the conventional route of electropolish, vacuum fire, glow discharge cleaning and in situ bake out. Glow discharge cleaning techniques have been accomplished over the past twenty years, in paticular for particle accelerator. Glow discharge cleaning has some permanent beneficial effects. It can produce near atomically clean surfaces on technical materials. There is a possibility of minimizing amount of beam conditioning time necessary to obtain full current operation and increase vacuum stability. Many experiments have shown that surfaces cleaned by glow discharge may be exposed to air for several weeks without serious recontamination. This is very important for an in situ untreated ultra-high vacuum system.

Considerable vacuum requirement of the Main Injector, especially for stable beam operation in the dynamic status, GDC can be applied either as a surface treatment prior to beam pipe assembly or in situ. In situ conditioning is much more efficient in quick recovery after installation or accidental air inrush. There is a possibility to practise electrodeless ECR discharge cleaning in situ conditioning technique for the Main Injector when needed.

As the cost of dipole magnet gap is very high, in situ bake out would rather not be adequate for beam pipes. The provision of an 1000°C vacuum furnace capable of accommodating large vacuum chamber is costly installation whereas the setting up of a gas discharge facility requires only standard vacuum equipments, a suitable D.C. power supply and a source of gas. It is very easily to practise for beam pipes. This will potentially provide an avenue for cost saving. Therefore the glow discharge cleaning technique seems to offer some very attractive features for the vacuum system of the Main Injector.